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(19) (CA) **CANADIAN PATENT (12)**

(54) **Process for Flocculating Recycle Water from Oil Sands Processing to Effect Efficiencies**

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ABSTRACT

In the process of recovering bitumen from oil sands wherein the oil sands are subjected to a hot water extraction comprising the steps of conditioning the oil sands, settling the conditioned oil sands in separation cells and subjecting the middlings layer to a scavenging step, the improvement of flocculating the discharge from the separation cells and/or scavenger circuits and recycling the released water to the conditioning step and separation cells.

1301692

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PROCESS FOR FLOCCULATING RECYCLE WATER FROM OIL SANDS PROCESSING TO EFFECT PROCESS EFFICIENCIES

BACKGROUND OF THE INVENTION

This invention relates to an improvement in the processing of recycle water from oil sands operations wherein bitumen is recovered from tar sands which are subsequently converted to petroleum products.

In the hot water extraction process of oil sands (also known as tar sands) in the northeast province of Alberta, Canada, the extraction method comprises three major process steps plus a final extraction used to clean up the recovered bitumen for further processing. In the first step, called conditioning, oil sand is mixed with water and heated with open steam to form a pulp of 70-85 wt.% solids. Sodium hydroxide or other reagents are added as required to maintain the pH in the range of about 8.0-8.5. In the second step, called separation, the conditioned pulp is diluted further with hot water so that separation can take place. The bulk of the sand-sized particles (greater than 325 mesh screen) rapidly settles and is withdrawn as sand tailings. Most of the bitumen rapidly floats (settles upward) to form a coherent mass known as bitumen froth which is recovered by skimming the separation vessel. An aqueous middlings layer containing some mineral and bitumen is formed between these layers. A scavenger step may be conducted in the middlings



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layer from the primary separation step to recover additional amounts of bitumen therefrom and this step usually comprises aerating the middlings. The froths recovered from the primary and the scavenger step can be combined, diluted with naphtha and centrifuged to remove water and minerals. The naphtha is then distilled for further processing. Hot water processes are described in Canadian Patent Numbers 882,668; 866,226; 891,472; 892,548; and 973,500. Tailings can be collected from the aforementioned processing steps and generally will contain solids as well as dissolved chemicals. The tailings are collected in a retention pond in which additional separation occurs. The tailings can also be considered as processing water containing solids which are discharged from the extraction process. The tailings comprise water, both the natural occurring water and added water, bitumen and mineral.

Conditioning tar sands for the recovery of bitumen consists of heating the oil sand/water mixture to process temperature (180°-200°F.), physically mixing the pulp to uniform composition and consistency, and the consumption (by chemical reaction) of the caustic or other added reagents. Under these conditions, bitumen is stripped from the individual sand grains and mixed into the pulp in the form of discrete droplets of a particle size on the same order as that of the sand grains. During conditioning, a large fraction of the clay particles becomes well dispersed and mixed throughout the pulp. The conditioning process which

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prepares bitumen for efficient recovery during the following process steps also causes the clays to be the most difficult to deal with in the tailings disposal operation.

The other process step, called separation, is actually the bitumen recovery step, the separation having already occurred during conditioning. The conditioned oil sand pulp is screened to remove rocks and unconditionable lumps of tar sands and clay. The reject material, "screen oversize," is discarded. The screened pulp is further diluted with water to promote two separation processes. Globules of bitumen, essentially mineral-free, float upward to form a coherent mass of froth on the surface of the separation units; and, at the same time, mineral particles, particularly the sand size material, settle down and are removed from the bottom of the separation unit as sand tailings. These two settling processes take place through a medium called the middlings. The middlings consist primarily of water, bitumen particles, and suspended fines.

The particle sizes and densities of the sand and of the bitumen particles are relatively fixed. The parameter which influences the separation processes most is the apparent viscosity of the middlings. Characteristically, as the suspended material content rises above a certain threshold, which varies according to the composition of the suspended fines, apparent viscosity rapidly achieves high values with the effect that the separation processes essentially stop. Little or no bitumen is recovered and all streams exiting the

1301692

unit have about the same composition as the feed. As the feed suspended fines content increases, more water must be used in the process to maintain middlings viscosity within the operable range.

The third step of the hot water process is scavenging. The feed of suspended fines content determines the process water requirement through the need to control middlings viscosity which, as noted before, is governed by the clay/water ratio and the type of clay minerals. It is usually necessary to withdraw a drag stream of middlings to maintain the separation unit material balance, and this stream of middlings can be scavenged for recovery of incremental amounts of bitumen. Air flotation is an effective scavenging method for this middlings stream.

Final extraction or froth clean-up is usually accomplished by centrifugation. Froth from primary extraction is diluted with naphtha, and the diluted froth is then subjected to a two stage centrifugation. This process yields an oil product of essentially pure, but diluted, bitumen. Water and mineral and any unrecovered bitumen removed from the froth constitutes an additional tailing stream which must be disposed.

In the terminology of extractive processing, tailings are a throwaway material generated in the course of extracting the valuable material from the non-valuable material. In oil sands processing, tailings consist of the whole oil sands plus net additions of process water less only

1301692

the recovered bitumen product. Oil sand tailings can be subdivided into three categories: (1) screen oversize; (2) sand tailings--the fraction that settles rapidly, and (3) middlings--the fraction that settles slowly. Screen oversize is typically collected and handled as a separate stream.

Tailings disposal is the operation required to place the tailings in a final resting place. Because the tailings contain bitumen emulsions, finely dispersed clay with poor settling characteristics and other contaminants, water pollution considerations prohibit discarding the tailings into rivers, lakes, or other natural bodies. Currently, the tailings are stored in retention ponds which involve large space requirements and the construction of expensive enclosure dikes. A portion of the clear water layer at the top of the tailings pond may be recycled back into the water extraction process as an economic measure to conserve water. Currently, two main operating modes for tailings disposal are: (1) dike building--hydraulic conveying of tailings followed by mechanical compaction of the sand tailings fraction; and (2) overboarding--hydraulic transport with no mechanical compaction.

The present invention is directed to the water in the tailings from the separation cells and/or the scavenging circuits which, in accord with the invention, is recycled back into the water extraction process and provides for improved process efficiencies and improved process economics.

1301692

SUMMARY OF THE INVENTION

The process of the invention involves flocculating the discharge from the separation cells and/or the scavenger circuits employed in the oil sands processing and returning to the conditioning drums and separation cells water recovered from the flocculated tailings. As a result of the process of the invention, water conservation, energy savings, increased bitumen recovery, and other benefits, as will be detailed later, are obtained.

DESCRIPTION OF THE DRAWINGS

Fig. 1 is a schematic flow diagram of water use in the oil sands extraction process as presently practiced.

Fig. 2 is a schematic flow diagram showing the flocculating step of the invention and recycle of the water from the flocculated tailings to the extraction process.

Fig. 3 is a schematic flow diagram showing a further embodiment of the invention.

DETAILED DESCRIPTION OF THE INVENTION

Reference is made to Figure 1 which indicates the manner in which process water is presently used in the extraction

1301692

process. As can be seen from the diagram, process water enters the steam exchangers through line 11 for heat exchange purposes and then passes into the conditioning drums and then to the separation cells and the scavenger circuits. The waste water tailings from the separation cells (line 13) and scavenger circuits (line 15) is fed to a tailings sump from which it passes to a distributor which directs the water to the retention pond. The upper layer of water in the tailing pond or river water is the source of process water (line 11) and since this water is cold, usually from about 40° to about 65°F., it must be heated before entering the conditioning drums.

In the process of the invention, however, as shown in Figure 2, the waste water from the scavenger circuits is fed through line 17 to one or more flocculating tanks. These tanks, usually cylindrical, hold the waste water while flocculating agents are added. Useful agents are any of the numerous and well known flocculating agents, but a particularly preferred agent is high molecular weight (4 to 6 million) water soluble, anionic polyacrylamide which has about 26% to about 36% of its amide groups hydrolyzed to carboxy groups and, preferably, converted to the sodium salt. This polyacrylamide has been characterized by Chemical Abstracts under Registry No. 37224-28-5 and is commercially available from Dow Chemical Company as SEPARAN™ AP273 polymer (see also U.S. 3,965,708 and Balakrishnan et al., AICHE J. 21(6), 1225-7). Another preferred flocculant is

# 1301692

PERCOL™ 1017 which is a medium high molecular weight anionic flocculant available from Allied Colloids Company. Of course, it will be understood that injection of the flocculant in tailings lines as well as other techniques may be used instead of direct addition to the flocculating tank.

The effect of the flocculant is, of course, to effect a flocculation of the inorganic solids in the waste water. The flocculated inorganic material settles to the bottom of the tank and is taken to a tailings sump, as shown, and then disposed to the distributor for direction to the retention pond. The bitumen rises to the top of the tank and is taken through line 25 and combined with the bitumen froth from the separation cells and scavenger circuits. The released water which rises to the top section of the tank is essentially free of the solids and is recycled through line 19, as shown, to the steam exchangers and then to the conditioning drums. The tailings water from the flocculating tank (line 21) is taken to a tailings sump where it is combined with the tailings from the separation cells (line 23).

Another embodiment of the invention is shown in Figure 3. As can be seen, in this procedure, the tailings from the separation cells and the scavenger circuits (whole tailings) are combined and the combined tailings in line 29 are injected with flocculant and fed to the flocculating tank. This procedure provides excellent mixing of the flocculant and better mixing of the sand from the separation cells and middlings from the scavenger circuits. This

1301692

technique is very cost efficient in that it reduces the capital expense for piping. It is desirable to construct the flocculating tank with two concentric weirs, the upper weir enabling the bitumen froth to spill over for transport through line 25, and the second, lower weir being used to collect the released water for transport through line 19.

A desirable embodiment of the invention is to employ a plastic or other suitable vessel as the flocculating tank which is floated in the retention pond. The bitumen and released water are simply pumped from the weirs at the top of the plastic container and the settled solids allowed to drop into the pond through the bottom of the vessel.

The amount of flocculant which is added to the waste tailings need only be sufficient to effect flocculation of the solids and, in general, this will be dosages from about 2.5 to about 100 ppm. It will be understood, of course, that the actual dosage to be used will vary with the particular flocculant employed and with the particular characteristics of the tails, but such parameters are readily determined by the skilled art worker.

Preference for use of the SEPARANT<sup>TM</sup> AD273 flocculant is based on the fact that it is extremely effective in making a separation of solids from the bitumen and water and, in fact, this flocculant can cause about 70% or more of the bitumen in the waste water to float on the recovered water's surface. The process of the invention provides numerous benefits which include a very high recovery of the bitumen heretofore lost

1301692

to tails. In addition, there is reduced cost of pumping tailings and recycle water and reduced sludge accumulation and associated reclamation costs. This results from the fines in the bottom of the flocculating tank entrapping the sand present and this effectively eliminates the formation of sludge, thus making the waste water more easily transported and more environmentally acceptable. Still another major advantage of the process is the heat savings obtained from the use of the hot recycle water (line 19) fed to the extraction system. Since this recycle water is at a temperature between about 120° to about 140°F., it significantly reduces the amount of steam needed to heat the additional cold make-up water for the process which comes from the tailings pond or river.

The following Table illustrates the benefits obtained from the process of the invention. The Table is based on calculations which show the savings obtained over a ten-year period using present production levels as a base line at a price of \$20.00 (Cdn.) per barrel for bitumen.

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**TABLE**

**ESTIMATED DOLLAR SAVINGS USING A FLOCCULATING TREATMENT  
FOR SCAVENGER TAILINGS IN AN OIL SANDS PROCESSING PLANT**

	<u>\$ x 10<sup>6</sup></u>
Increased Bitumen Recovered:	252
Reduced Cost of Natural Gas for Steam and Boilers:	7
Reduced pumping costs:	<u>1</u>
Total	260
Less Flocculant Costs:	<u>32</u>
Net Savings:	228

1301692

CLAIMS

Claim 1. In the process of recovering bitumen from oil sands wherein the oil sands are subjected to a hot water extraction comprising the steps of conditioning the oil sands, settling the conditioned oil sands in separation cells and subjecting the middlings layer to a scavenging step, the improvement of flocculating the inorganic material in the discharge from the separation cells and/or the scavenger circuits and recycling the released water to the conditioning step.

Claim 2. The process of Claim 1 wherein the discharge from the scavenging circuits is flocculated.

Claim 3. The process of Claim 2 wherein the flocculant employed is a high molecular weight (4 to 6 million) water soluble, anionic polyacrylamide which has about 26% to about 36% of its amide groups hydrolyzed to carboxy groups.

Claim 4. The process of Claim 3 wherein the flocculant is employed at a dosage of from about 2.5 to about 100 ppm.

Claim 5. In the process of recovering bitumen from oil sands wherein the oil sands are subjected to a hot water extraction comprising the steps of

conditioning the oil sands, settling the conditioned oil sands in separation cells and subjecting the middlings layer to a scavenging step, the improvement of flocculating the inorganic matter in the combined discharge from the separation cells and scavenger circuits and recycling the released water to the conditioning step.

**Claim 6.** The process of Claim 5 wherein the flocculating agent is employed in an amount of from about 2.5 to 100 ppm.

**Claim 7.** The process of Claims 5 or 6 wherein the flocculant is a high molecular weight (4 to 6 million) water soluble, anionic polyacrylamide which has about 26% to about 36% of its amide groups hydrolyzed to carboxy groups.

**Claim 8.** The process of Claim 7 wherein flocculant is in the form of sodium salt.

**Claim 9.** The process of Claims 1, 2, 3, 4, 5, 6 or 8 wherein the flocculant is the polyacrylamide characterized by Chemical Abstracts Registry No. 37224-28-5.

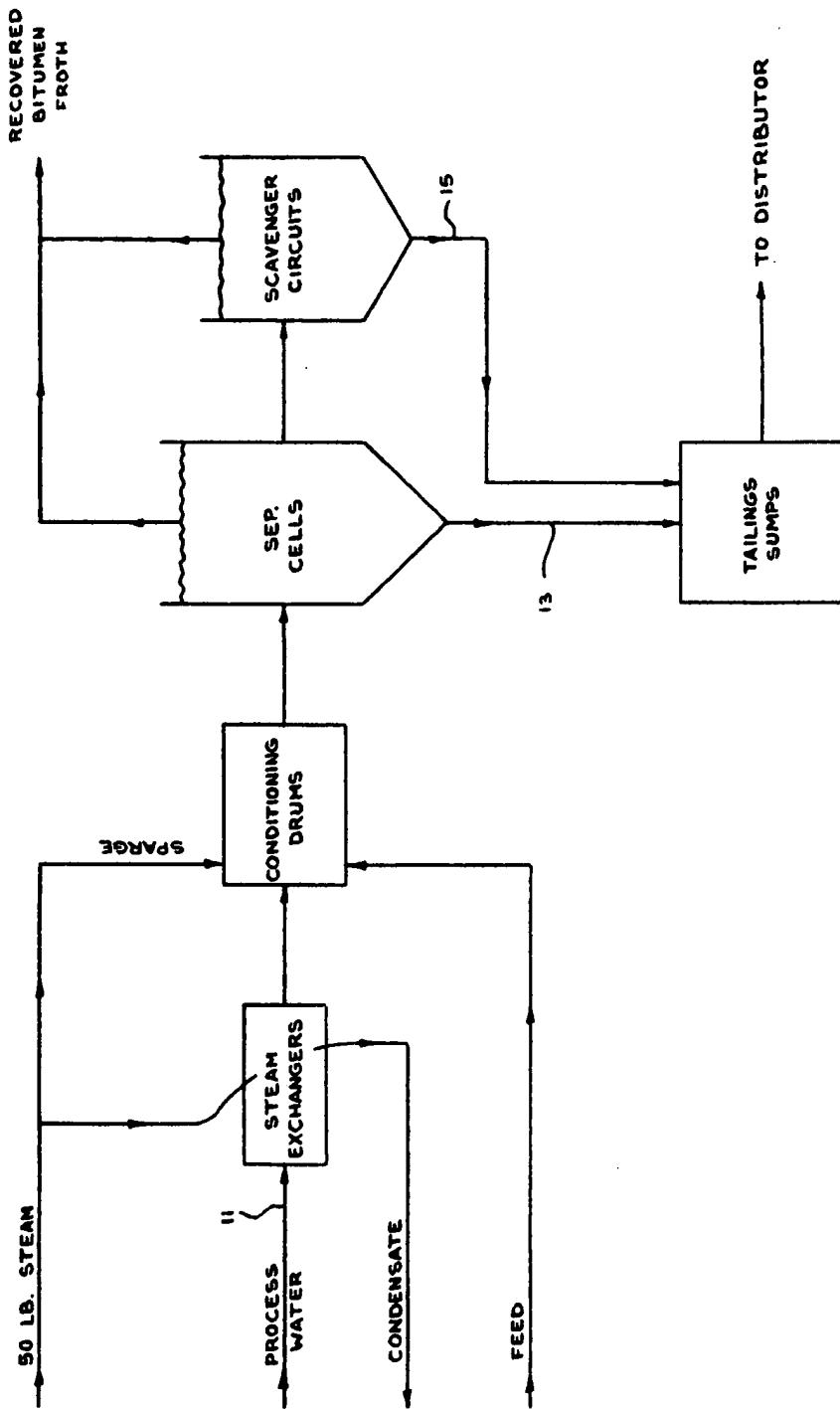
**Claim 10.** The process of Claims 1, 2, 3, 4, 5, 6 or 8 wherein the flocculation is made to occur in a tank floating in a retention pond for waste tailings.



1301692

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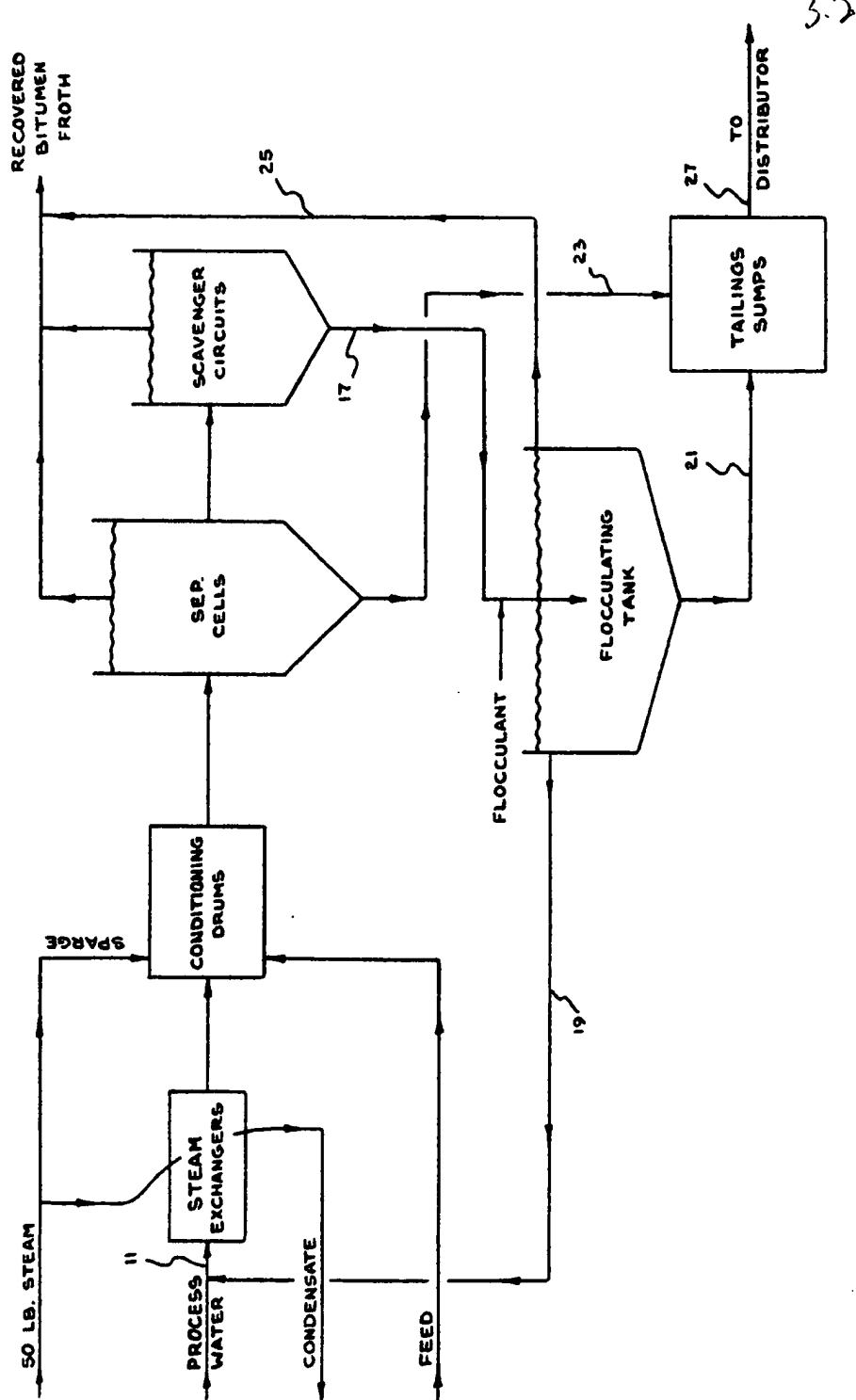
FIG. 1  
SCHEMATIC FLOW DIAGRAM OF WATER USE IN PRIMARY OIL SANDS  
EXTRACTION UNDER PRESENT CONDITIONS



Gawling & Henderson

1301692

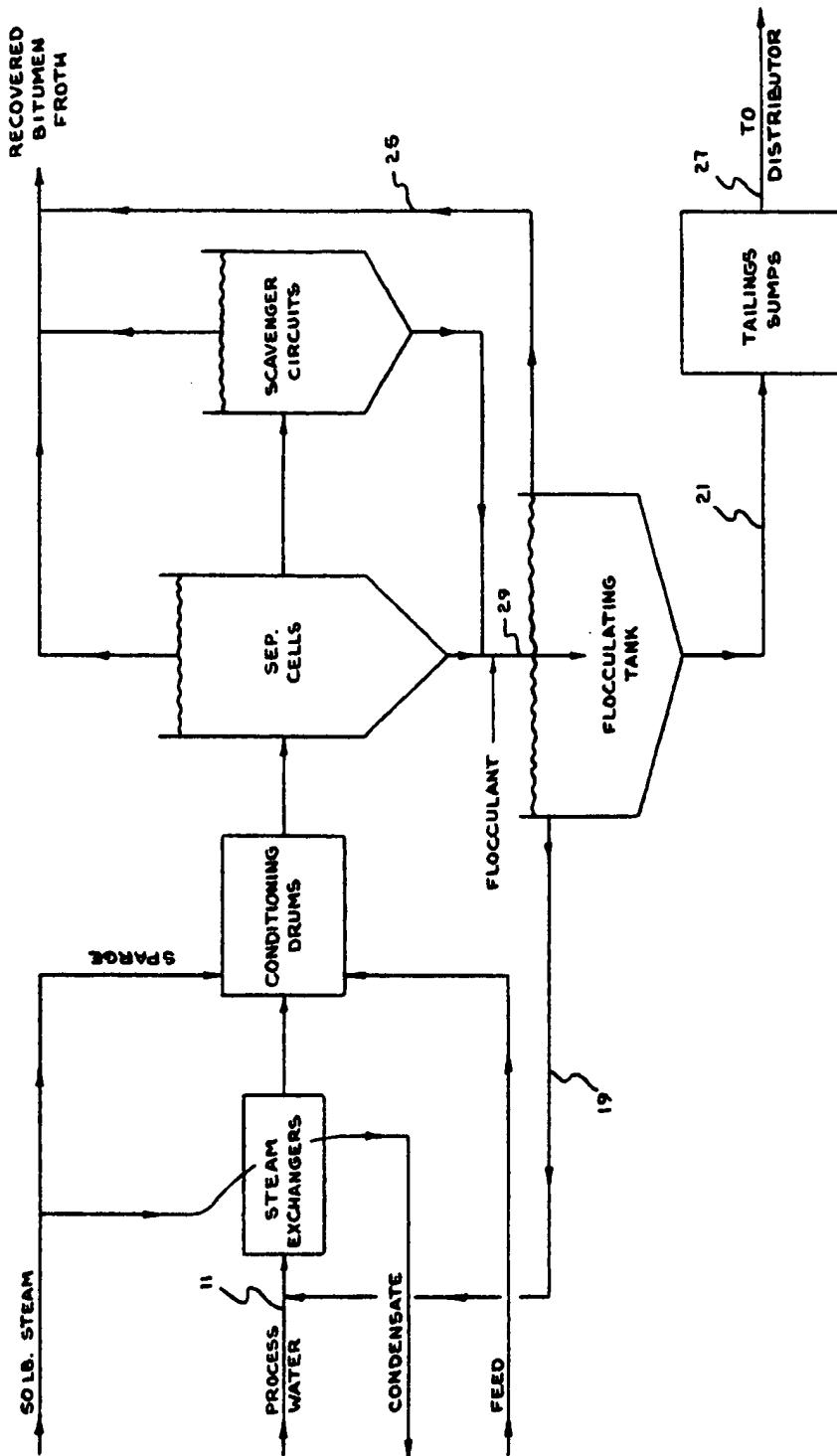
FIG. 2  
SCHEMATIC FLOW DIAGRAM OF WATER USE IN PRIMARY OIL SANDS  
EXTRACTION OF FLOCCULATED TAILINGS



1301692

3-3

FIG. 3  
SCHEMATIC FLOW DIAGRAM OF WATER USE IN PRIMARY OIL SANDS EXTRACTION  
OF FLOCCULATED TAILINGS - WHOLE TAILINGS ALTERNATE



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